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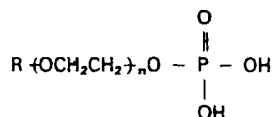
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(54) Conveyor track lubricant composition employing phosphate esters and method of using same.

(57) It has been found that a conveyor system can be lubricated with dilute [ $1.0 \times 10^{-3}M$ ] aqueous solutions of a partially neutralized mono phosphate ester of the formula:



wherein n has a value 0 to 3 and R is a C<sub>12</sub> to C<sub>20</sub> saturated or partially unsaturated linear alkyl group, or a mixture of such groups. The use of a synergist such as a long-chain alcohol, a fatty-acid derived amine oxide, or urea improves the properties of the lubricant compositions.

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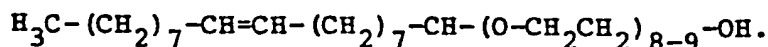
BACKGROUND OF THE INVENTION

The present invention relates generally to water-based lubricant compositions and a method of using such compositions. More particularly, the invention  
5 discloses aqueous compositions containing a long-chain phosphate ester which are useful for the lubrication of continuously-moving conveyor systems, particularly conveyor systems used in the packaging of materials intended  
10 ed for human consumption.

Continuously-moving conveyor systems employed in food and beverage packaging operations require both cleansing and lubrication to insure that the systems function properly in moving items between packaging  
15 stations. For example, in those conveyor systems employed in bottling and capping operations, a clean and properly lubricated conveyor surface permits stoppage of the bottles during the filling and capping operations while the conveyor track continues to move underneath.  
20 If the track is not clean or does not have the proper lubricity, the containers may be knocked over or fail to stop moving, resulting both in bottle breakage and in disruption of the bottling line. These cleansing and lubricating functions are customarily accomplished  
25 by circulating an aqueous dispersion or solution of a water-based material across the track surface.

Various surface active agents have been employed as lubricants in food packaging processes, including long chain fatty acid soaps, sulfonated oils, and alkanolamides. Of particular interest to the present invention is the water-soluble agent of U.S. Patent 3,574,100 which claims a track lubricant composition comprising a mixture of an imidazoline derivative and an alkali metal salt of the phosphate esters of oleyl alcohol ethoxylate.

The specific phosphate ester salt disclosed in the 3,574,100 patent is a material manufactured by General Aniline and Film Corporation under the trade name, Gafac GB520. Gafac GB520 is the partial sodium salt of a mixture of mono- and di-alkyl phosphate esters derived from an oleyl alcohol polyethoxylate having eight to nine ethoxy groups, together with 35 percent of unreacted oleyl alcohol ethoxylate, i.e.,

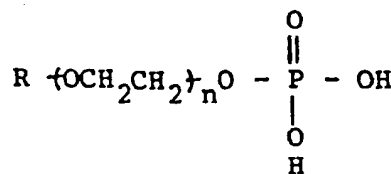


The mole ratio of phosphate diester to phosphate monoester in the Gafac GB520 material is greater than 3 to 1.

#### DESCRIPTION OF THE INVENTION

The novel lubricants of the present invention are aqueous compositions, at least partially neutralized with a base, preferably comprising two active components. The first of these active ingredients is a mono alkyl phosphate ester of the formula

35



5 The second of the active ingredients is a synergist taken from the group:

A) an alcohol of the formula



B) an amine oxide of the formula



C) urea.

20 The lubricant compositions also contain minor amounts of di alkyl phosphate ester and phosphoric acid. In the above formulae R and R' are linear saturated primary alkyl groups, C<sub>14</sub> through C<sub>18</sub>, or linear partially unsaturated primary alkylene groups, C<sub>16</sub> through C<sub>20</sub>. R and R' can also comprise a mixture of saturated or unsaturated alkyl groups C<sub>12</sub> through C<sub>20</sub>, having an average chain length in the range C<sub>13</sub> through C<sub>18</sub>. R is a mixture of saturated and unsaturated alkyl groups C<sub>8</sub> through C<sub>18</sub> having an average chain length C<sub>12</sub> through C<sub>18</sub> (i.e., products derived from fatty acids such as 30 coco oil and tallow). The value of m and n can be zero through three.

We have discovered that while lubricating compositions employing phosphate monoesters as the sole active ingredient exhibit good lubricity, the use of an 35 alcohol, an amine oxide, or a urea synergist with the phosphate ester greatly improves the performance of the

lubricant composition, particularly in the area of durability. Although the addition of other materials, such as surfactants, can improve the lubricant formulations, excellent compositions can be prepared wherein the sole active components comprise a neutralized aqueous dispersion of a two-component system containing a monophosphate ester and one of the synergist compositions described above.

These lubricant compositions exhibit excellent lubricity at concentrations as low as .01 percent. The use of higher concentrations, (i.e., .05 to .1 percent) is generally preferred, however, because it results in greater durability for the applied material.

The phosphate esters of the present invention can be prepared in a conventional manner. For example, a  $C_{14} - C_{15}$  triethoxyphosphate was synthesized by reacting a  $C_{14} - C_{15}$  linear primary alcohol mixture (Neodol<sup>R</sup> 45) with three moles of ethylene oxide and then phosphorylating the resultant ethoxylate with polyphosphoric acid at a temperature of approximately 65-80° C. The product comprised monophosphate ester, unreacted ethoxylated alcohol, phosphoric acid, and diester. Other methods of obtaining phosphate esters are illustrated in the prior art, i.e., U.S. Patents 1,970,578, 2,174,271, 2,167,326 and 3,033,889.

It is generally desirable to limit the formation of diester product in the reaction mixture. The diesters are approximately twice the molecular weight of the monoesters and do not add to the system's lubricating properties. Thus, based on cost considerations, the diester content should be kept to a minimum, preferably less than 15 percent of the active mono ester ingredient. This can be accomplished by employing an excess of phosphoric acid in the reaction. If desired, additional amounts of long-chain alcohol or alcohol ethoxylate can be added to the product after cooling.

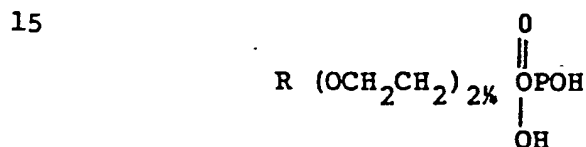
In view of the fact that the compositions of the present invention typically contain free phosphoric acid as well as the acidic monophosphate ester, the lubricant compositions would have an undesirably low (acid) pH were they not partially neutralized. Moreover, it has been found that neutralization results in better dispersibility of the compositions in water. Neutralization can be accomplished by the addition of any suitable base, but the use of  $\text{NH}_3$ ,  $\text{NH}_4\text{OH}$ , or a basic aminofunctional material is generally preferred.

Although the lubricity of the ester compositions remains relatively constant over a range of pH values, the durability of the composition peaks at a pH of 5-6, the pK range for neutralization of the first hydroxyl group in mono alkyl phosphate esters. If the neutralization is continued to a pH = 9 (the pK range for the second hydroxyl neutralization), there is a fall-off in product durability. Thus optimum properties in terms of durability are obtained with partial neutralization, whereby the resultant composition has a pH in the range 5.0 to 7.0, ideally pH = 6.0. However, good lubricity can be obtained with compositions of higher pH values -- in the range 6.5 to 10, preferably 7.0 - 10.0. Some applications may warrant a partial sacrifice of product durability in order to obtain the benefit of a mildly basic lubricant. For example, formulations having a basic pH can resist acidic beverage spills (e.g., beer) without any major deterioration in lubricity. Moreover, basic (pH 8.0 to 10.0) compositions are generally desirable where it is necessary to incorporate a sequestering agent in the lubricant to overcome problems resulting from water hardness.

As previously indicated, the long chain saturated alkyl or partially unsaturated alkylene substituent groups of the phosphate esters may contain up to

three ethoxy groups in the chain. The presence of these groups increases the dispersibility of the ester in water but at some sacrifice in lubricity. Therefore, while the lubricant performance of a typical  
 5 monophosphate ester increases with the length of the alkyl chain, saturated alkyl groups longer than C<sub>15</sub> tend to be too insoluble to be easily formulated, absent some degree of ethoxylation. Increasing the ethylene oxide content increases solubility but reduces  
 10 lubricating ability.

A preferred ester representing a compromise between lubricating performance and solubility (for saturated R groups) is the phosphate ester,



20 where R is a 50:50 mixture of C<sub>14</sub>H<sub>29</sub> -- and C<sub>15</sub>H<sub>31</sub> -- alkyl groups.

The performance of the lubricant compositions is improved by the presence of a free long-chain alcohol, a long-chain alcohol ethoxylate, a fatty-acid-  
 25 derived amine oxide, or urea. Although these additives provide no lubricity by themselves, their presence in the lubricating formulation serves as a synergist, improving both the lubricity and, in particular, the durability of the compositions. While even a minor amount of these additives serves to improve the properties of  
 30 the lubricants, optimum results are obtained when the long-chain monoester/synergist ratio is in the range of 1:1.5 on a molar basis.

If an alcohol is used as the synergist, the  
 35 alcohol can be the reactant precursor of the phosphate

ester, and thus R and R' will be the same. However, this identity is not necessary and desirable lubricant compositions can be formulated utilizing various combinations of alcohol and phosphate ester which differ  
5 either in the length or in the nature (saturated/unsaturated, ethoxylated/non-ethoxylated) of the linear long-chain substituant group.

The amine oxide synergist employed in certain species of the invention is an ethoxylated polyoxyethylene fatty amine, characteristically derived from a  
10 natural product such as coco oil, tallow, soybean oil, and the like. Particularly useful compounds include bis(2-hydroxyethyl) cocoamine oxide and bis(2-hydroxyethyl) tallow amine oxide.

15 The inclusion of a sequesterant such as ethylenediamine tetra acetic acid (EDTA) in the lubricant composition renders the phosphate ester formulations resistant to the formation of insoluble precipitates when used in hard water formulations.

20 The phosphate-ester-containing lubricants are generally applied at very low levels of concentration, i.e., between about  $1.0 \times 10^{-3}$  and  $3.0 \times 10^{-3}$  M, based on the monophosphate ester. It is therefore desirable to supply the lubricants in the form of liquid concentrates which can be further diluted with water prior to  
25 use. Concentrates containing about 10 percent by weight of monophosphate ester can be formulated by blending the phosphate ester, synergist and other desired ingredients at elevated temperatures (30-50°C) in an isopropanol/water mixture and neutralizing to a pH of 5.0-6.5  
30 with a base. Lower molecular weight phosphate esters so formulated will remain stable at room temperature. Higher molecular weight concentrates may solidify when cooled to ambient temperature, but can be re-liquified  
35 by heating to 30-40°C. The concentrates are diluted



prior to use, typically in the ratio one part concentrate to 100 to 200 parts water.

5 The compositions are applied to the surface of moving conveyor systems so that the surface of the conveyor does not become dry. This may be accomplished by passing the conveyor through a dip or trough containing a dilute solution of the lubricant, by brushing or roller coating the lubricant composition on the surface of the conveyor, or by other conventional  
10 means. The preferred method of application, however, utilizes spray nozzles spaced along the conveyor track to ensure the proper degree of lubricity. No matter how the material is applied, it is important that the conveyor system remain consistently wet.

15 As noted above, the lubricant compositions may be improved by use of surfactants and/or sequesterants. Preferred surfactants are long-chain anionic materials such as the sodium salt of sulfonated oleic acid.

20 Should the lubricant concentrate be diluted with hard water, a highly-dispersed suspension may form, giving the product a cloudy appearance. The fine suspension may in time agglomerate and accumulate in the spray nozzles, causing them to block. The use of a  
25 sequestering agent is desirable to control the hardness. In view of the fact that sequestering agents such as EDTA perform most efficiently at pH of 8.0 or more, it may be desirable to supply the lubricant concentration at a sufficiently high pH (8.0 + 10) to  
30 enable the sequestering agent to operate at maximum efficiency. As has been previously mentioned, however, this increase in pH may result in some loss in the product's durability.

The lubricity and durability of the lubricant  
35 compositions reported in the following examples was

measured on a pilot test track, approximately 19 cm wide by 305 cm long. The track, a slat conveyor manufactured of 304 stainless steel, was operated at a speed of 40 cm/sec. Friction measurements were measured with a 0-5 pound precision load cell manufactured by Transducers, Inc. (Model C462). The output from the load cell was connected to a Sigma variable speed strip chart recorder, permitting measurements of coefficient of friction ( $\mu$ ) vs. time.

A constant load, consisting of eight filled bottles connected to the load cell by a loop of fine wire, was utilized in the experiments. Lubricant compositions were applied in spurts to the test track with a feed pump, via a fan spray nozzle. The pump and nozzle could be adjusted to vary both the frequency of the spurts and the volume of material delivered. In each of the following examples a spurt rate of 36/minute and a solution volume of 32 mL/minute was employed.

The test was initiated by activating the feed pump and starting the test track and recorder in motion. The measured coefficient of friction was observed to drop over a period of time to a constant equilibrium value ( $\mu$ ), the value depending on the lubricity of the applied material. Because lubricity has an inverse relationship to friction, the lower the value of  $\mu$ , the better the lubricant.

The pilot test track was also used to measure the durability of the lubricant compositions. This was accomplished by replacing the intermittent stream of lubricant applied to the track with a constant stream of water. At the same time, a weighted fiberglass cloth (5000 gms, width 11 cm) was placed on the track to increase the wear rate. The time required for the coefficient of friction to increase from the lubricated

equilibrium value to the original friction value represents the durability of the test lubricant. The greater the time, the better the durability.

5       The reported data for coefficients of friction represent measurements taken five minutes after the initial activation of the feed pump and test track to ensure that a constant equilibrium had been obtained. Immediately after making this measurement, the flow of lubricant was replaced with a water stream and the durability test was commenced.

10       In interpreting the following data, the optimum products are those which exhibit both low equilibrium coefficients of friction and high durability times. Except as otherwise indicated, all formulations were diluted in distilled water to  $2.2 \times 10^{-3}$  M based on the weight of the ester. All ratios are expressed in terms of weight, unless otherwise indicated. Finally, although the following examples illustrate the present invention, they should not be construed as limiting the invention to their details.

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30

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TABLE I

PHOSPHATE ESTER 2.2 X 10 <sup>-3</sup> M	RATIO OF ESTER TO SECONDARY COMPONENT(S)	PILOT TRACK PERFORMANCE DUR/μ
Ex 1A: C <sub>12</sub> -C <sub>15</sub> OPO <sub>3</sub> H <sub>2</sub>	Isolated ester	8 min/0.134
Ex 1B:	1:1.3 <sup>†</sup> C <sub>12</sub> -C <sub>15</sub> alcohol	10 min/0.145
Ex 2: C <sub>12</sub> -C <sub>15</sub> (EtO) <sub>3</sub> OPO <sub>3</sub> H <sub>2</sub>	1:1 C <sub>12</sub> -C <sub>15</sub> (EtO) <sub>3</sub> OH	6 min/0.147
Ex 3A: C <sub>14</sub> -C <sub>15</sub> OPO <sub>3</sub> H <sub>2</sub>	Before separation (1:1.1 C <sub>14</sub> -C <sub>15</sub> OH)	16.5/0.12
Ex 3B:	Isolated ester	6/0.132
Ex 3C:	1:0.5 C <sub>14</sub> -C <sub>15</sub> OH	15/0.121
Ex 3D:	1:1 C <sub>14</sub> -C <sub>15</sub> OH	17/0.112
Ex 3E:	1:1 C <sub>14</sub> -C <sub>15</sub> (EtO) <sub>2</sub> OH	16/0.127

<sup>†</sup> All ratios are expressed in terms of weight of ester (1) to weight of added ingredient.

TABLE I  
(Continued)

PHOSPHATE ESTER 2.2 X 10 <sup>-3</sup> M	RATIO OF ESTER TO SECONDARY COMPONENT (S)	PILOT TRACK PERFORMANCE DUR/ $\mu$
Ex 3F:	1:1 C <sub>16</sub> (EtO) <sub>10</sub> OH	4/0.136
Ex 3G:	1:1 C <sub>18</sub> OH	27/0.104
Ex 3H:	1:0.5 C <sub>18</sub> OH	13.5/0.123
Ex 4A: Oleyl(EtO) <sub>2</sub> OPO <sub>3</sub> H <sub>2</sub>	Before separation (1:1.2 Oleyl (EtO) <sub>2</sub> OH)	16/0.123
Ex 4B:	Isolated ester	10/0.12
Ex 4C:	1:0.25 Oleyl (EtO) <sub>2</sub> OH	13/0.122
Ex 4D:	1:0.5 Oleyl (EtO) <sub>2</sub> OH	14/0.13
Ex 4E:	1:1 Oleyl (EtO) <sub>2</sub> OH	14.5/0.128

TABLE I  
(Continued)

PHOSPHATE ESTER 2.2 X 10 <sup>-3</sup> M	RATIO OF ESTER TO SECONDARY COMPONENT	As received (esters plus 35% Oleyl (Eto) <sub>9</sub> OH)	PILOT TRACK PERFORMANCE DUR/μ
Ex 5: Gafac GB520 [(Oleyl (Eto) <sub>9</sub> O) <sub>2</sub> PO <sub>2</sub> H:] Oleyl (Eto) <sub>9</sub> OPO <sub>3</sub> H <sub>2</sub> (molar ratio dl to mono - 4:1 molar)			3/0.22
Ex 6A: C <sub>14</sub> -C <sub>15</sub> (Eto) <sub>2</sub> OPO <sub>3</sub> H <sub>2</sub>	Ester: C <sub>14</sub> -C <sub>15</sub> (Eto) <sub>2</sub> OH: Sulfonate OA-5 (sodium salt of sulfonated oleic acid) 1:1:0.5		15.5/0.136*

\* Dispersed in 150 ppm tap water.

TABLE I  
(Continued)

PHOSPHATE ESTER 2.2 X 10 <sup>-3</sup> M	RATIO OF ESTER TO SECONDARY COMPONENT	PILOT TRACK PERFORMANCE DUR/μ
Ex 6B:	Ester: C <sub>14</sub> -C <sub>15</sub> (EtO) 2% OH: Sulfonate OA-5 1:1:1	18/0.136 (fresh soln) *
	Ester: C <sub>14</sub> -C <sub>15</sub> (EtO) 2% OH: Sulfonate OA-5 1:1:1	13/0.14 (4* day old soln)
Ex 6C:	Ester: C <sub>14</sub> -C <sub>15</sub> (EtO) 2% OH: Sulfonate OA-5 1:0.5:0.5	15/0.145 *

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\* Dispersed in 150 ppm tap water.

TABLE I  
(Continued)

PHOSPHATE ESTER 2.2 X 10 <sup>-3</sup> M	RATIO OF ESTER TO SECONDARY COMPONENT	PILOT TRACK PERFORMANCE DUR/μ
Ex 6D:	Ester: C <sub>14</sub> -C <sub>15</sub> (EtO) 2% OH: Sulfonate OA-5 1:0.1:0.5	15/0.151*
Ex 7: C <sub>14</sub> -C <sub>15</sub> (EtO) 2% OPO <sub>3</sub> H	Aromox C/12 (bis (2-hydroxy ethyl) cocoamine oxide) 1:1	19/0.143*
Ex 8A: C <sub>14</sub> -C <sub>15</sub> (EtO) 2% OPO <sub>3</sub> H	Urea 1:0.8	19/0.15

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\* Dispersed in 150 ppm tap water.



TABLE I  
(Continued)

PHOSPHATE ESTER 2.2 X 10 <sup>-3</sup> M	RATIO OF ESTER TO SECONDARY COMPONENT	PILOT TRACK PERFORMANCE DUR/μ
Ex 8B: C <sub>14</sub> -C <sub>15</sub> (EtO) <sub>2</sub> OPO <sub>3</sub> H	Urea 1:2	29/0.15
Ex 9A: Oleyl (EtO) <sub>4</sub> OPO <sub>3</sub> H <sub>2</sub> (50:50 mono:di by weight)	Before separation (1:0.1 Oleyl (EtO) <sub>4</sub> OH)	12/0.145
Ex 9B:	1:1 Igepal CO-530	8/0.158
Ex 9C:	1:1 C <sub>18</sub> OH	13.5/0.136
Ex 9D:	1:1 Oleyl (EtO) <sub>2</sub> OH	12/0.158
Ex 10A: Tallow (EtO) <sub>2</sub> OPO <sub>3</sub> H <sub>2</sub>	Before separation (51% Tallow (EtO) <sub>2</sub> OH)	16/0.125
Ex 10B:	Isolated ester	9/0.12

TABLE I  
(Continued)

PHOSPHATE ESTER 2.2 X 10 <sup>-3</sup> M	RATIO OF ESTER TO SECONDARY COMPONENT	PILOT TRACK PERFORMANCE DUR/μ
Ex 10C:	1:1 C <sub>14</sub> -C <sub>15</sub> (Eto) 2% OH	15/0.12
Ex 10D:	1:1 C <sub>18</sub> OH	15/0.12
Ex 10E:	1:1 Stearyl (Eto) 2% OH	16/0.13

Example I $C_{12}$ - $C_{15}$  non-ethoxylated monoester

5           A phosphated ester was prepared by reacting a  
           $C_{12}$  to  $C_{15}$  linear primary alcohol mixture with  
          polyphosphoric acid at a temperature of approximately  
          72°C. Upon cooling the product was analyzed and found  
10          to contain 38.2 percent monoester, 11.5 percent  $H_3PO_4$ ,  
          50.3 percent unreacted alcohol, and no measurable di-  
          ester.

          The phosphate ester was separated from the  
          unreacted alcohol by dissolving approximately 25 g of  
          the above product in 50 mL of warm isopropyl alcohol  
15          and stirring the mixture with heating until a clear  
          solution was obtained. The pH of the solution was  
          adjusted to approximately 6.5 by the addition of con-  
          centrated  $NH_4OH$  (dilute solutions should be avoided due  
          to the ester's solubility in  $H_2O$ ). This resulted in  
20          the formation of a white precipitate which was digested  
          at 40°C for 45 minutes. The solution was cooled to  
          20°C, filtered through a buchner funnel, washed with  
          cold IPA, and the precipitate dried. The residue --  
          the ammonium salts of the phosphate ester and polyphos-  
25          phoric acid -- was dissolved in water to form a  $2.2 \times 10^{-3}$  M  
          lubricant composition based on the phosphate  
          ester, and applied to the test track in the manner pre-  
          viously described. The durability and lubricity of the  
          composition was measured and recorded. The results are  
30          set forth at Ex 1A in Table I.

          A second sample of the phosphate ester/unreact-  
          ed alcohol product of the first reaction was formulated  
          into a lubricant without removal of the unreacted alcohol.  
          This was accomplished by adding 25 g of material (38  
35          parts ester: 50 parts alcohol) to 250 ml of a 10/90

IPA/H<sub>2</sub>O solution, and heating the mixture to 35°C, with stirring. The pH of the dispersion was adjusted to 6.5 by the addition of diethanol amine (DEA), and then diluted with distilled water to form a  $2.2 \times 10^{-3}$  M

5 lubricant composition based on the phosphate ester. The composition was applied to the test track and its durability and lubricity measured and recorded. The results are set forth at Ex 1B in Table I.

10

Example II

C<sub>12</sub>-C<sub>15</sub> triethoxylated monoester

A commercially-available phosphated ester  
15 composition (Alkaphos-3, Alkaril Chemicals Ltd.), was formulated into a track lubricant composition. Analysis of the product as received revealed it to be a C<sub>12</sub>-C<sub>15</sub> triethoxyphosphate and that its composition was 45 percent monoester, 10 percent phosphoric acid, 40 percent unreacted alcohol ethoxylate, and 5 percent  
20 diester.

Twenty-five (25) grams of the product was dispersed in 250 ml of a 10/90 IPA/H<sub>2</sub>O solution, neutralized to pH 6.5 with DEA, and diluted to  $2.2 \times 10^{-3}$   
25 M with distilled water in the same manner as the ester/alcohol material of Example 1. The results of the composition as a track lubricant are set forth at Ex 2 of Table 1.

30

Example III

C<sub>14</sub>C<sub>15</sub> non-ethoxylated monoester

A phosphated monoester was prepared from a  
35 C<sub>14</sub>C<sub>15</sub> linear primary alcohol mixture by following the procedure of Example 1. Analysis revealed the product

to contain 41.8 percent monoester, 10.7 percent  $H_3PO_4$ , 47.5 percent unreacted alcohol, and no measurable di-  
 ester. A portion of the product was dissolved in 30/70  
 IPA/ $H_2O$ , neutralized to pH 6.5 with DEA, diluted to  
 5  $2.2 \times 10^{-3}$  M in distilled water and tested on the pilot  
 track. The results appear at Ex 3A of Table I.

Another portion of the above product was treat-  
 ed so as to isolate the phosphate ester from the non-  
 reacted alcohol in accordance with the separation step  
 10 of Example 1. The isolated, partially neutralized ester/  
 acid mixture was tested on the pilot track by itself  
 (Ex 3B), and in combination with a number of long-chain  
 alcohols and alcohol ethoxylates (Exs 3C-3H). In all  
 cases, the  $C_{14}$ - $C_{15}$  phosphate ester-containing lubricant  
 15 was diluted to  $2.2 \times 10^{-3}$  M, based on the ester content,  
 prior to testing.

#### Example IV

##### 20 Oleyl diethoxy monoester

An oleyl alcohol diethoxylate was prepared by  
 reacting oleyl alcohol  $[CH_3(CH_2)_7CH=CH(CH_2)_7CHOH]$  with  
 ethylene oxide in a 1 to 2 molar ratio. The reaction  
 25 was carried out by charging the oleyl alcohol and a  
 sodium hydroxide catalyst into an autoclave, heating  
 the autoclave to  $140^\circ C$ , and gradually adding ethylene  
 oxide gas. The pressure was maintained at 25 to 30  
 p.s.i. during the reaction.

The resultant oleyl alcohol diethoxylate was  
 30 reacted with polyphosphoric acid by following the pro-  
 cedure of Example 1. Analysis revealed the product to  
 be 42 percent phosphate monoester, 8 percent  $H_3PO_4$ , and  
 50 percent alcohol ethoxylate diester. A portion of  
 35 the product was dissolved in 30/70 IPA/ $H_2O$ , neutral-  
 ized to pH 6.5 with DEA, diluted to  $2.2 \times 10^{-3}$  M in

distilled water and tested on the pilot track. The results appear at Ex 3A of Table I.

Another portion of the product was treated so as to isolate the phosphate ester from the alcohol in accordance with the separation step of Example 1. The isolated, partially neutralized ester/acid mixture was tested on the pilot track by itself (Ex 4B), and in combination with a number of long-chain alcohols and alcohol ethoxylates (Exs 4C-4E). In all cases the oleyl diethoxy phosphate-ester-containing lubricant was diluted to  $2.2 \times 10^{-3}$  M based on the ester content, prior to testing.

#### Example V

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#### Oleyl polyethoxy ester (Gafac GB520)

A commercially-available partially neutralized ethoxylated oleyl alcohol ester composition (Gafac GB520, GAF, Inc.) was formulated into a track lubricant. Analysis of the product as received revealed the material to be an aqueous dispersion containing a long-chain phosphate diester, a long-chain phosphate monoester and free oleyl alcohol ethoxylate. The analysis showed the product to be partially neutralized with NaOH.

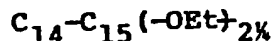
The degree of ethoxylation of the oleyl alcohol alkylene substituent was determined to be between 8 and 9, and the ratio of phosphate diester to phosphate monoester to be in excess of 4 to 1. Unreacted ethoxylated alcohol was found to constitute approximately 35 percent by weight of the solids ingredients.

The GB520 was formulated into a  $2.2 \times 10^{-3}$  M track lubricant composition, based on the weight of the total ester. The results of the pilot track test utilizing this material are reported at Ex 5 of Table 1.

As evident from these results, the composition exhibited poor durability and lubricity.

Example VI

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phosphate monoester plus surfactant

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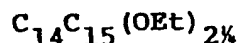
A lubricant composition was formulated wherein a surfactant was added to improve the hard-water stability of the composition. A phosphate ester of a  $C_{14}-C_{15}(EtO)_2$  alcohol ethoxylate was prepared in accordance with the procedure of Example IV. Analysis revealed the product to be 72.5 percent monoester, 9 percent diester, 12 percent phosphoric acid, and 6.5 percent unreacted alcohol ethoxylate.

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An anionic surfactant -- the sodium salt of sulfonated oleic acid, 50% active (Sulfonate OA5, Cities Service, Inc.) -- was added to the ester/alcohol blend in various amounts, the mixture neutralized to a pH of 6.5 with DEA, and the neutralized products dispersed in 150 ppm tap water to form track lubricants ( $2.2 \times 10^{-3}$  M, based on ester). The results are recorded at Exs 6A-6D in Table I.

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Example VII



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phosphate monoester plus amine oxide

The  $C_{14}C_{15}(OEt)_2$  alcohol ethoxylate of Example VI was treated so as to isolate the mono-phosphate ester in accordance with the separation procedure of Example I. Ten Ten (10) grams of the monoester was

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dispersed in 75 grams of 2:13 IPA:H<sub>2</sub>O, and titrated with NH<sub>4</sub>OH to a pH of 6.0. Five (5) grams of Aromox C/12, bis(2-hydroxyethyl) cocoamine oxide (Arma Industrial Chemicals), was added to the above solution to form a concentrate, and the material dispersed in tap water (100:1) to form a track lubricant ( $2.2 \times 10^{-3}$  M, based on ester). The results are recorded at Ex 7 of Table I.

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Example VIII

C<sub>14</sub>-C<sub>15</sub>(OEt)<sub>2</sub>%

phosphate monoester plus urea

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The C<sub>14</sub>C<sub>15</sub>(OEt)<sub>2</sub>% alcohol ethoxylate of Example VI was treated so as to isolate the mono-phosphate ester in accordance with the separation procedure of Example I. Ten (10) grams of the monoester was dispersed in 80 grams of 1:8 IPA:H<sub>2</sub>O and titrated with NH<sub>4</sub>OH to a pH of 6.0. A concentrate was formulated by dissolving 8 grams of urea in the above solution. The concentrate was diluted in distilled water (100:1) to form a track lubricant. The results are reported at EX 8A of Table I.

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A second lubricant composition was prepared, as above, but employing a higher level of urea (20 grams). The results are recorded at Ex 8B of Table I.

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Example IX

Long chain alkylene phosphate esters were formulated into track lubricant compositions in accordance with the procedures of Example 1 through VIII.

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The results of the test of these compositions on a pilot test track are summarized in Table I, Exs 9 and 10.

Example X

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Effect of pH

10 In order to test the effect of pH on lubricant properties, a track lubricant concentrate was formulated as follows:

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15	Phosphate Ester	<div> <div> C<sub>14</sub>-C<sub>15</sub> triethoxy monophosphate ester 80% </div> <div> C<sub>14</sub>-C<sub>15</sub> triethonyl alcohol (nonionic) 8% </div> <div> phosphoric acid (free) 12% </div> </div>	8.0
	Nonionic Alcohol		1.0
	Isopropanol		12.0
20	Urea		15.0
	Water (soft)		60.4
	Ethylene Diamine Tetra Acetic Acid Disodium.2H <sub>2</sub> O		1.0
	Methanolamine (MEA) or KOH*		2.6

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\*The amount and nature of the base was altered to obtain different pH's. With 2.6% MEA, pH = 8.0.

30 Track lubricant compositions were prepared from concentrates having a range of pH from 8.0 through 11.0, and applied to the test track in the manner previously described. The lubricity and durability of these compositions are set forth in Table II.

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TABLE II

	<u>Example</u>	<u>pH Concentrate Formula</u>	<u>pH 1:250 Soft Water Dilution</u>	<u>Durability</u>	<u>μ</u>
5	A	8.0	7.6	6.50	0.145
	B	9.0	8.3	6.50	0.147
	C	10.0	9.2	5.50	0.155
	D	10.0 (KOH)	9.2	4.01	0.147
	E	11.0 (KOH)	9.3	3.21	0.142

10 It can be noted that the coefficient of friction ( $\mu$ ) stays relatively constant with increase in pH, and that durability decreases with further increase in pH. In certain applications, i.e., where the lubricant is applied substantially constantly to the conveyor

15 track, a relatively low-level of durability may be satisfactory.

Example XI

20 Resistance to beverage spills

The resistance of the lubricant compositions to acidic beverage spills such as those generally found in beer bottling halls was tested utilizing the formula-

25 tion of Example X, partially neutralized with MEA and thereafter diluted with soft water (250:1) to a pH of 7.8.

Forty beer bottles were placed on the clean pilot test track and the test track apparatus adjusted as previously outlined. In an effort to simulate a

30 heavily used track surface as found in a commercial bottling facility, the track was operated at a speed of 40 cm/sec. for a period of two hours with lubricant supplied throughout at a rate of 41 mL/minute.

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At the end of the two hour equilibration period, with the track and the lubricant-feed still running, the effect of eight test bottles interconnected with a precision load cell was measured for 4½ minutes. The equilibrium coefficient of friction was found to be  $\mu = 0.154$ .

After the above equilibrium measurement was completed, the flow of lubricant to the track was supplemented with a spray of water to determine the effect of dilution on the lubricant composition. Water was sprayed onto the track through a nozzle, upstream of the eight-bottle load, at a rate of 103 mL/minute using a peristaltic pump. Measurements were taken over a 4½ minute period and the water-dilution coefficient of friction was found to be  $\mu = 0.159$ . Flow of water was stopped and the coefficient of friction was again measured over a period of 4 minutes:  $\mu = 0.159$ .

The effect of a beer spill was determined by applying a supplemental spray of beer (pH 4.1) to the track in the same manner as the water spray in the preceding water-dilution step, but at a rate of 55 mL/minute. The pH of the combined beer and lubricant spray on the track was determined to be pH = 4.5. The coefficient of friction, measured over a 7 minute period, was observed initially to rise ( $\mu = 0.167$ ) and then to fall ( $\mu = 0.159$ ). Thereafter, the spray of beer to the test track was stopped and the lubricant alone applied. The lubricity was found to increase somewhat ( $\mu = 0.152$ ).

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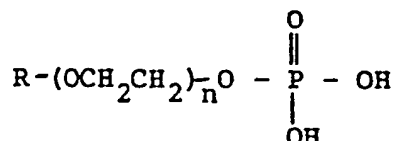
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WHAT IS CLAIMED:

1. In a method for lubricating a continuously-moving conveyor system for transporting packages from a first station to a second station and wherein said packages are temporarily detained at said first station while said conveyor moves beneath said packages, and said conveyor system is wetted with an aqueous lubricant composition;

the improvement wherein said aqueous lubricant composition comprises:

a mono alkyl phosphate ester of the formula



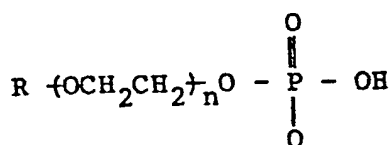
wherein the concentration of phosphate ester in said lubricant composition is in the range  $1.0 \times 10^{-3}$  to  $3.0 \times 10^{-3}$  molar and the weight of mono alkyl phosphate ester exceeds the weight of any dialkylphosphate ester, and wherein n can have a value of zero through three, and R is selected from the group consisting of i) linear saturated primary alkyl groups,  $\text{C}_{14}$  through  $\text{C}_{18}$ , ii) linear partially unsaturated primary alkyl groups  $\text{C}_{16}$  through  $\text{C}_{20}$ , and iii) a mixture of linear primary alkyl substituents  $\text{C}_{12}$  through  $\text{C}_{20}$ , saturated or partially unsaturated, wherein the average length of the alkyl substituent is  $\text{C}_{13}$  through  $\text{C}_{18}$ ; and

said lubricant composition being at least partially neutralized with a base whereby its pH is in the range 5.0 through 10.0.

2. In a method for lubricating a continuously-moving conveyor system for transporting packages from a first station to a second station and wherein said packages are temporarily detained at said first station while said conveyor moves beneath said packages, and said conveyor system is wetted with an aqueous lubricant composition;

the improvement wherein said aqueous lubricant composition comprises:

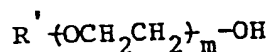
A. a mono alkyl phosphate ester of the formula



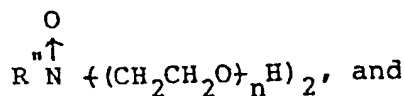
wherein the concentration of phosphate ester in said lubricant composition is in the range  $1.0 \times 10^{-3}$  to  $3.0 \times 10^{-3}$  molar and the weight of mono alkyl phosphate ester exceeds the weight of any dialkylphosphate ester;

B. An synergist selected from the group consisting of:

i) an alcohol of the formula



ii) an amine oxide of the formula



iii) urea,

said synergist present in an amount between 10 and 150 percent of the weight of said monoester;

wherein m and n can have a value of zero through three, R and R' are selected from the group consisting of i) linear saturated primary alkyl groups, C<sub>14</sub> through C<sub>18</sub>, ii) linear partially unsaturated primary alkyl groups C<sub>16</sub> through C<sub>20</sub>, and iii) a mixture of linear saturated primary alkyl groups C<sub>12</sub> through C<sub>20</sub>, wherein the average length of the alkyl substituant is C<sub>13</sub> through C<sub>18</sub>, and R'' is a mixture of saturated and unsaturated alkyl groups C<sub>8</sub> through C<sub>18</sub> having an average chain length C<sub>12</sub> through C<sub>18</sub>; and

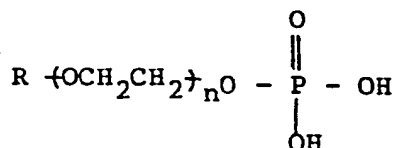
said lubricant composition being at least partially neutralized with a base whereby its pH is in the range 5.0 through 10.0.

3. A method according to Claim 1 or 2 further including:

applying said lubricant compositions to said conveyor by means of a plurality of spray nozzles spaced along said conveyor system.

4. An aqueous lubricant composition comprising:

A. a mono alkyl phosphate ester of the formula



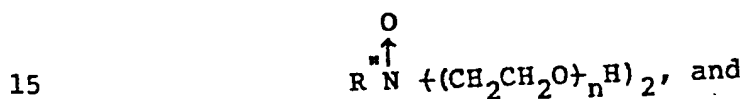
wherein the concentration of phosphate ester in said lubricant composition is in the range  $1.0 \times 10^{-3}$  to  $3.0 \times 10^{-3}$  molar and the weight of mono alkyl phosphate ester exceeds the weight of any dialkylphosphate ester;

5 B. An synergist selected from the group consisting of:

i) an alcohol of the formula



ii) an amine oxide of the formula



iii) urea,

20 said synergist present in an amount between 10 and 150 percent of the weight of said monoester;

wherein m and n can have a value of zero through three, R and R' are selected from the group consisting of i) linear saturated primary alkyl groups, 25  $C_{14}$  through  $C_{18}$ , ii) linear partially unsaturated primary alkyl groups  $C_{16}$  through  $C_{20}$ , and iii) a mixture of linear saturated primary alkyl groups  $C_{12}$  through  $C_{20}$ , wherein the average length of the alkyl substituant is  $C_{13}$  through  $C_{18}$ , and R'' is a mixture 30 of saturated and unsaturated alkyl groups  $C_8$  through  $C_{18}$  having an average chain length  $C_{12}$  through  $C_{18}$ ; and

said lubricant composition being at least partially neutralized with a base whereby its pH is in 35 the range 5.0 through 10.0.

5. An aqueous lubricant composition according to Claim 4 wherein said base is selected from the group consisting of ammonia, ammonium hydroxide, or a water soluble amine.

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6. An aqueous lubricant composition according to Claim 4, further including an anionic surfactant.

7. The composition of Claim 6 wherein said  
10 surfactant is the sodium salt of sulfonated oleic acid.

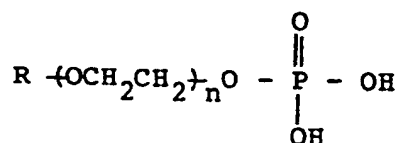
8. An aqueous lubricant composition according to Claim 4, further including a sequestering agent.

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9. An aqueous lubricant concentrate comprising:

A. a mono alkyl phosphate ester of the formula  
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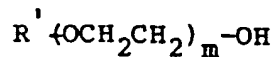
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wherein the concentration of phosphate ester in said concentrate is in the range 0.2 to 0.5 molar and the weight of mono alkyl phosphate ester exceeds the weight of any dialkyl phosphate ester.

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B. An synergist selected from the group consisting of:

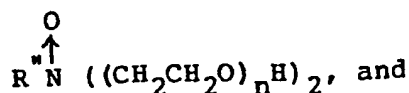
i) an alcohol of the formula



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ii) an amine oxide of the formula





5                   iii) urea,

said synergist present in an amount between 10 and 150 percent of the weight of said monoester;

10                   wherein m and n can have a value of zero through three, R and R' are selected from the group consisting of i) linear saturated primary alkyl groups, C<sub>14</sub> through C<sub>18</sub>, ii) linear partially unsaturated primary alkyl groups C<sub>16</sub> through C<sub>20</sub>, iii) a mixture  
15 of linear saturated primary alkyl groups C<sub>12</sub> through C<sub>20</sub>, wherein the average length of the alkyl substituant is C<sub>13</sub> through C<sub>18</sub>, and R'' is a mixture of saturated and unsaturated alkyl groups C<sub>8</sub> through C<sub>18</sub> having an average chain length C<sub>12</sub> through C<sub>18</sub>; and  
20                   said lubricant concentrate being at least partially neutralized with a base whereby its pH is in the range 5.0 through 10.0.

25                   10. An aqueous lubricant concentrate according to Claim 9 wherein said base is selected from the group consisting of ammonia, ammonium hydroxide, or a water soluble amine.

30                   11. An aqueous lubricant concentrate according to Claim 9, further including an anionic surfactant.

35                   12. The composition of Claim 11 wherein said surfactant is the sodium salt of sulfonated oleic acid.

13. An aqueous lubricant concentrate according to Claim 9, further including a sequestering agent.

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European Patent  
Office

# EUROPEAN SEARCH REPORT

0137057

Application number

EP 83 10 9715

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 7)
A	DE-A-2 313 330 (BASF WYANDOTTE)  * Claim; page 2, paragraph 2 - page 7, paragraph 4 *	1,2,4 6-9,11 -13	C 10 M 3/04 C 10 M 3/40
D,A	US-A-3 574 100 (C.A. WETMORE) * Claim 1 *	1	
A	FR-A-1 426 694 (MOBIL OIL) * Claims A,B1,2,4 *	1	
A	US-A-3 404 090 (R.J. BETTY) * Column 2, lines 16-52 *	1,2	
			TECHNICAL FIELDS SEARCHED (Int. Cl. 7)
			C 10 M
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 19-04-1984	Examiner RO TSAERT L.D.C.
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</p>			